TECHNICAL REPORT RK-83-4

LIQUID CHROMATOGRAPHIC ANALYSIS OF NITROCELLULOSE-BASE PROPELLANTS - FINAL REPORT OF FIRST YEAR EFFORT

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28 MARCH 1983

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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

A procedure has been developed that is able to predict the optimum conditions for separation of the ingredients of a complex mixture by liquid chromatography. The procedure involves establishing a data base of retention times for a large number of propellant ingredients in 7 predetermined combinations of 3 solvents. When a new combination of some of these ingredients is encountered, a computer analysis of the retention times of the ingredients from the data base will predict the best solvent composition to separate all the ingredients. The details of the procedure and several examples of its use are discussed.

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ABSTRACT - Continued

This project has been accomplished as part of the US Army Materials Testing Technology Program, which has for its objective the timely establishment of testing techniques, procedures, or prototype equipment (in mechanical, chemical or nondestructive testing) to insure efficient inspection methods for materiel/material procured or maintained by DARCOM.

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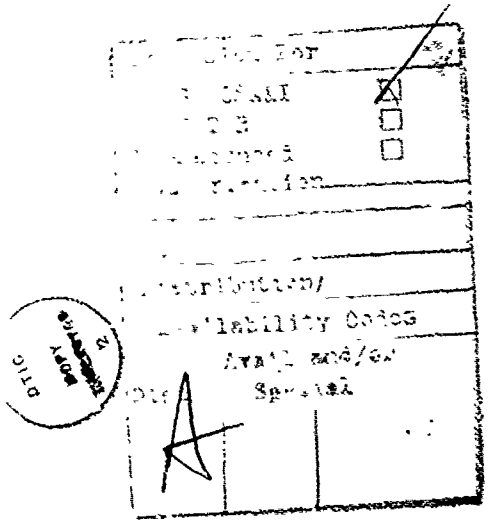

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I. INTRODUCTION

Nitrocellulose-based propellants are widely used throughout the military and civilian areas in guns and rockets. There are almost as many different procedures for analysis as there are propellants. Even in the area of liquid chromatography, frequently, more than one procedure is used in the analysis of the propellant. Most techniques have been designed to answer a specific question about the purity or amount of two or three ingredients. However, most propellants have at least three or four original ingredients and frequently one or two other reaction products that can be easily extracted.

Since these ingredients can be extracted into a solvent it would seem logical to try to analyse for all them at once. A major difficulty to this in the past has been that silica columns are difficult to work with. Precise control of water, alcohols, and other polar ingredients is necessary to obtain reproducible retention times and amounts. Reverse phase columns-octadecylsilane or C-18 do not have this problem but investigators have expressed difficulty in obtaining good separations of more than a few ingredients with the binary mixtures commonly used.

Recently some significant interest has been observed in using more than 2 solvent systems with C-18 columns. Manufacturers have started producing HPLC equipment that can pump 3 or 4 solvents simultaneously. The reason for a multi-solvent system is that separations are accomplished by more than differences in polarity. Snyder[1] has proposed that solvents should be classified as to three properties - proton acceptor, proton donor, and dipole interaction as indicated in Figure 1. It is believed that these properties make the major contributions to separations in HPLC. Kirkland[2] has developed a procedure consisting of 7 experiments which can be used to predict the best solvent composition for separating ingredients in a mixture. A program was undertaken to develop a procedure that will separate the maximum number of commonly used propellant ingredients possible.

II. EXPERIMENTAL

A. Equipment

All solvents were obtained from Burdick and Jackson Laboratories. Prior to use the solvents were filtered and degassed in an ultrasonic bath. Water was distilled and deionized by a MILLI-Q system from Millipore. The liquid chromatographic system consisted of three M6000A pumps, a 720 system controller, a 710B WISP sample injector, and RCM 100 radial compression system, all from Water Associates. The column used was a Waters Associates C-18 radial PAC column using 10 micron silica coated with octadecyl silane. The detector was a LC 85 variable wavelength UV detector from Perkin-Elmer. Responses were most often monitored at 215 nm. The signal from the detector was analyzed by a Hewlett-Packard 3353E laboratory automation system.

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[1] Snyder, L. R. J Chrom Sci 16, (1978), 223.
[2] Glatch, J. L., Kirkland, J. J., Squire, K. M., and Miner, J. M.; J. Chromatgography, 199, (1980), 57.

B. Sample Preparation

The missile propellant was cut into small pieces and approximately 400 µg were accurately weighed out and placed in a test tube. To this was added an accurately weighed amount of diethylphthalate as an internal standard. Twenty-five ml of acetonitrile was added to the test tube. After one hour the contents of the test tube were subjected to ultrasonic vibrations using a Sonicator micro tip for 15 minutes. The solution was then filtered and placed in a WISP sample vial for analysis.

The gun powder was prepared in a similar manner except that the acetonitrile was diluted and there was no soak time. The reason from this modification was that the powder was not cross linked and tended to form a gum in pure acetonitrile. Usually 50% acetonitrile in water was sufficient to effect complete extraction.

III. PROCEDURE

There are numerous papers and books on ways to evaluate mixtures in general. The paper by Snee[3] presents a good summary of many of the techniques in a manner easily understood by the uninitiated. Examination of these methods in light of snyder's[1] paper permits development of an experimental procedure involving only 7 to 10 experiments to obtain a complete picture of the separation properties of the three solvents and their mixtures. Snyder[1] suggested the use of methanol, acetonitrile, and tetrahydrofuran as the three solvents of choice for reversed phase chromatography. In the real world we want a separation or resolution of the ingredients in a sample within a certain time limit. It has been shown[4] that resolution will increase with increasing $\frac{K}{1+k}$ but increases in values of K greater than 10 or 15 have only minimal influence on the resolution. The first step in developing a separation is to adjust the three solvents strengths by dilution with an inert carrier, such as water, until the retention times of all the ingredients in the sample are between 2 and 30 minutes (assuming non retained peaks elute at 1 minute). This produces 3 solvents of approximately the same strengths but greatly differing selectivities. Once the dilution of one solvent has been selected, the desired dilution of the other two can be roughly estimated as described by Kirkland[5,2]. For methanol, acetonitrile and tetrahydrofuran the following calculations were made:

$$\phi_a P_a + \phi_b P_b = \phi_A P_A + \phi_B P_B \qquad (1)$$

Where ϕ_A = % by vol of A

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[3] Snee, R. D.; Chemtech; Nov (1979) 702.

[4] "Introduction to Modern Liquid Chromatography", L. R. Snyder and J. J. Kirkland, Wiley, New York, 2nd edition, 1979, Ch 2.

[5] Reference 4, Chapter 6.

$P_{H_2O} = 10.2$ for ACN: 50%(5.8) + 50%(10.2) = 8.00

$P_{ACN} = 5.8$ for THF: 38%(4.0) + 64%(10.2) = 7.97

$P_{MEOH} = 5.1$ for MEOH: 43%(5.1) 57%(10.2) = 8.01

$P_{THF} = 4.0$

The dilutions finally selected were 60% methanol in water, 50% acetonitrile in water, and 42% tetrahydrofuran in water. For convenience these will be identified as MEOH, ACN, and THF.

At this point the 7 to 10 experiments suggested by Snee can be performed. Here the retention times of each ingredient are determined in each of the mixtures of the three solvents as shown in Table I. Only the first 7 are necessary to generate the response surface but mixtures 8, 9, and 10 can be used to check the accuracy of the analysis.

TABLE I. SOLVENT COMPOSITIONS

Experiment	1	2	3	4	5	6	7	8	9	10
MEOH	100	0	0	50	50	0	33.3	16.6	16.6	66.4
ACN	0	100	0	50	0	50	33.3	16.6	66.4	16.6
THF	0	0	100	0	50	50	33.3	66.4	16.6	16.6

Once the retention times have been obtained the data can be analyzed to develop a response surface for the separations. The resolution of any two ingredients can be calculated for any solvent mix of composition ($X_1A + X_2B + X_3C$) from equation (2)

$$R = B_1*X_1 + B_2*X_2 + B_3*X_3 + B_4*X_1*X_2 + B_5*X_1*X_3 + B_6*X_2X_3 + B_7*X_1*X_2*X_3 \quad (2)$$

The values for B_1 through B_7 are derived from equations 3 through 9.

Here Y_1 is the resolution of a peak pair in solvent mixture 1.

$$B_1 = Y_1 \quad (3)$$

$$B_2 = Y_2 \quad (4)$$

$$B_3 = Y_3 \quad (5)$$

$$B_4 = 4*Y_4 - 2(Y_1 + Y_2) \quad (6)$$

$$B_5 = 4*Y_5 - 2(Y_1 + Y_3) \quad (7)$$

$$B_6 = 4*Y_6 - 2(Y_2 + Y_3) \quad (8)$$

$$B_7 = 27*Y_7 - 12(Y_4 + Y_5 + Y_6) + 3(Y_1 + Y_2 + Y_3) \quad (9)$$

A computer program is being developed to perform these calculations and plot the response surface for selected resolutions of pairs of ingredients. The analysis is complicated by changes in order of elution of several ingredients as the solvent composition is varied. For this reason each ingredient in the mixture must be evaluated with each of the other ingredients for resolution.

IV. RESULTS

Samples of three gunpowders were obtained from a commercial manufacturer. A mixture was prepared that contained all of the ingredients and the probable reaction products. Table II lists the retention times of each ingredient for the 7 solvent mixtures suggested.

The B values calculated for several pairs of ingredients are listed in Table III.

Most of the ingredients are always resolved by a minimum value of 1.25 and are not considered critical to the separation. Figure 2 is a superimposed graph of the lines of resolution of the critical ingredients.

As can be seen in Figure 2 there is a small area of the graph where all ingredients can be resolved by the minimum value of 1.25. The solvent mixture in the middle of this area was selected and the 10 ingredient mixture was analysed. The resulting chromatograph, Figure 3, shows that indeed all the ingredients are well separated. At this point the three gun powder samples were prepared for analysis. Samples A and B did not contain Diphenylphthalate so this was selected as the internal standard. The internal standard for sample C was Dimethylphthalate since it already contained Diphenylphthalate. Chromatograms and averaged analysis of each sample are given in Figures 4, 5, and 6 and Tables IV, V, and VI.

A sample of cross linked double-base propellant was obtained for analysis. The individual ingredients were placed in 3 different mixtures to aid in identification and analysed by the same solvent scheme in Table I. The B coefficients for the critical peak pairs are given in Table VII and the superimposed response surfaces are given in Figure 7. Here a much larger area of solvent choice occurred. The propellant sample was prepared using Diethylphthalate as an internal standard. It was analysed using three different solvent mixtures within the indicated area and these are shown in Figures 8, 9, and 10.

The results of the analysis were in good agreement with other analyses of the propellant.

It is interesting to note that gas chromatography has difficulty in analysing for thermally labile ingredients, such as nitroglycerine, and low volatility materials, such as HMX. Liquid chromatograpny is generally limited to solubility and detectability of the ingredient. Nitroglycerine does not absorb well in the UV at 254 nm. To detect it one must go down to about 214 or 220 nm. HMX has a low solubility in most solvents. In the first sample

preparation of the propellant sample the HMX analysis was low while all other ingredients were accurate. Examination of the procedure disclosed that not enough solvent had been used to dissolve all the HMX. Subsequent preparation used larger amounts of solvent and the analysis improved.

V. CONCLUSIONS

The procedure described here seems to have almost universal application in liquid chromatography. Once the retention times for a list of ingredients is developed no further experimentation is required. When a new propellant composition is encountered, the solvent composition necessary for optimum separation of the ingredients can be quickly predicted with the aid of a computer.

A data base of 30 of the most commonly used propellant ingredients is being prepared for further testing. Currently a mixture of 10 ingredients can be separated with one solvent mixture. It is the ultimate goal that all 30 ingredients can be separated with one solvent mixture but it is doubtful that this can be achieved.

A variable that has not been examined yet is the influence of using different columns. During the next year this will be evaluated by using two C18 columns from the same manufacturer and other C18 columns from different manufacturers. It is expected that the within manufacturer variations will be small and not require changes in the optium solvent mixture. There will probable be significant differences between manufacturers but the extent of the differences can not be predicted at this time.

TABLE II. GUNPOWDER INGREDIENT RETENTION TIMES

SOLVENT MIX / INGREDIENTS	1	2	3	4	5	6	7
DMP	1 70	1 86	1 24	1.89	1.33	1.33	1.42
DEP	3.09	3.99	2 01	3.94	2.23	2.33	2.57
2, 4 ONT	3.35	3.34	1.91	3.15	2.15	2.66	2.90
NG	2.70	2.03	3.01	2.53	3.13	5.42	3.52
EC	5.61	16.5	3.70	13.20	4.10	9.40	6.28
NDPA	4.84	6.24	5.67	6.25	2.74	5.23	4.94
DPA	5.61	6.90	5.67	7.43	5.69	5.79	7.89
DPP	13.96	21.0	5 26	22.5	7.13	10.86	11 74
DBP	6.99	35	7.92	32	10.15	15	16

DMP = DIMETHYL PHTHALATE
DEP = DIETHYL PHTHALATE
2,4 DNT = 2,4 DINITRO TOLUENE
NG = NITROGYLCERINE
EC = ETHYL CENTRALITE
NDPA = N-NITROSODIPHENYLAMINE
DPA = DIPHENYLAMINE
DPP = DIPHENYLPHTHALATE
DBP = DIBUTYLPHTHALATE

TABLE III. GUNPOWDER B COEFFICIENTS FOR PAIRS OF INGREDIENTS

	B1	B2	B3	B4	B5	B7	B7
DMP-NG	6	1.02	10.62	1.32	9.96	74.88	-77.04
DEP-2,4 DNT	1.56	-3.9	-.6	-14.28	-3.84	16.92	83.52
2,4 DNT-NG	-3.9	-7.86	6.6	8.64	18.12	68.76	-139.68
NG-NDPA	12.84	25.26	15.96	13.08	-66.96	-87	166.14
EC-NDPA	-4.62	-61.56	11.82	-34.44	-47.04	-.6	518.4
EC-DPA	0	-57.6	11.82	-23.28	14.52	4.92	684.36
NDPA-DPA	4.62	3.96	0	11.16	61.56	5.52	165.96
DPA-DPP	50.1	84.6	-2.46	92.28	-60.72	-42.6	-533.34

TABLE IV. GUNPOWDER A ANALYSIS

Nitroglycerine	11.421 ± .057
DNT	.354 ± .001
NDPA	.327 ± .009
DPA	.652 ± .008
2-NDPA	.069 ± .001
DBP	1.980 ± .002

TABLE V. GUNPOWDER B ANALYSIS

Nitroglycerine	46.4 ± .7
DNT	.234 ± .002
NDPA	.294 ± .003
DPA	.054 ± .001
2-NDPA	.050 ± .000
DBP	.115 ± .004

TABLE VI. GUNPOWDER C ANALYSIS

Nitroglycerine	6.649 ± .007
DNT	.067 ± .006
NDPA	.264 ± .003
DPA	.243 ± .004
EC	.535 ± .001
2-NDPA	.098 ± .001
DPP	.592 ± .001
DEP	.043 ± .002

TABLE VII. B COEFFICIENTS FOR PROPELLANT FOR PAIRS OF INGREDIENTS

	B1	B2	B3	B4	B5	B6	B7
1-2 BTTN-NG	-3.24	-5.54	-12.72	-5.76	24.48	9.6	-20.7
1-6 BTTN-4-NDPA	-6.42	-3.12	-15.36	16.68	9.24	-55.92	-26.1
1-8 BTTN-DEP	-.9	6.36	-18.72	.12	10.2	-76.08	34.74
2-4 NG-N-MNA	-3.18	2.28	-2.64	22.44	-15.24	-65.52	-5.4
2-8 NG-DEP	2.34	11.76	-6.0	5.88	-14.28	-85.68	55.44
3-4 MNA-N-MNA	2.1	1.92	5.64	21.72	-7.56	-2.88	-85.14
3-8 MNA-DEP	7.62	11.4	2.28	5.16	-6.6	-23.04	-24.3
4-8 N-MNA-DEP	5.52	9.48	3.36	16.56	.96	-20.16	60.84

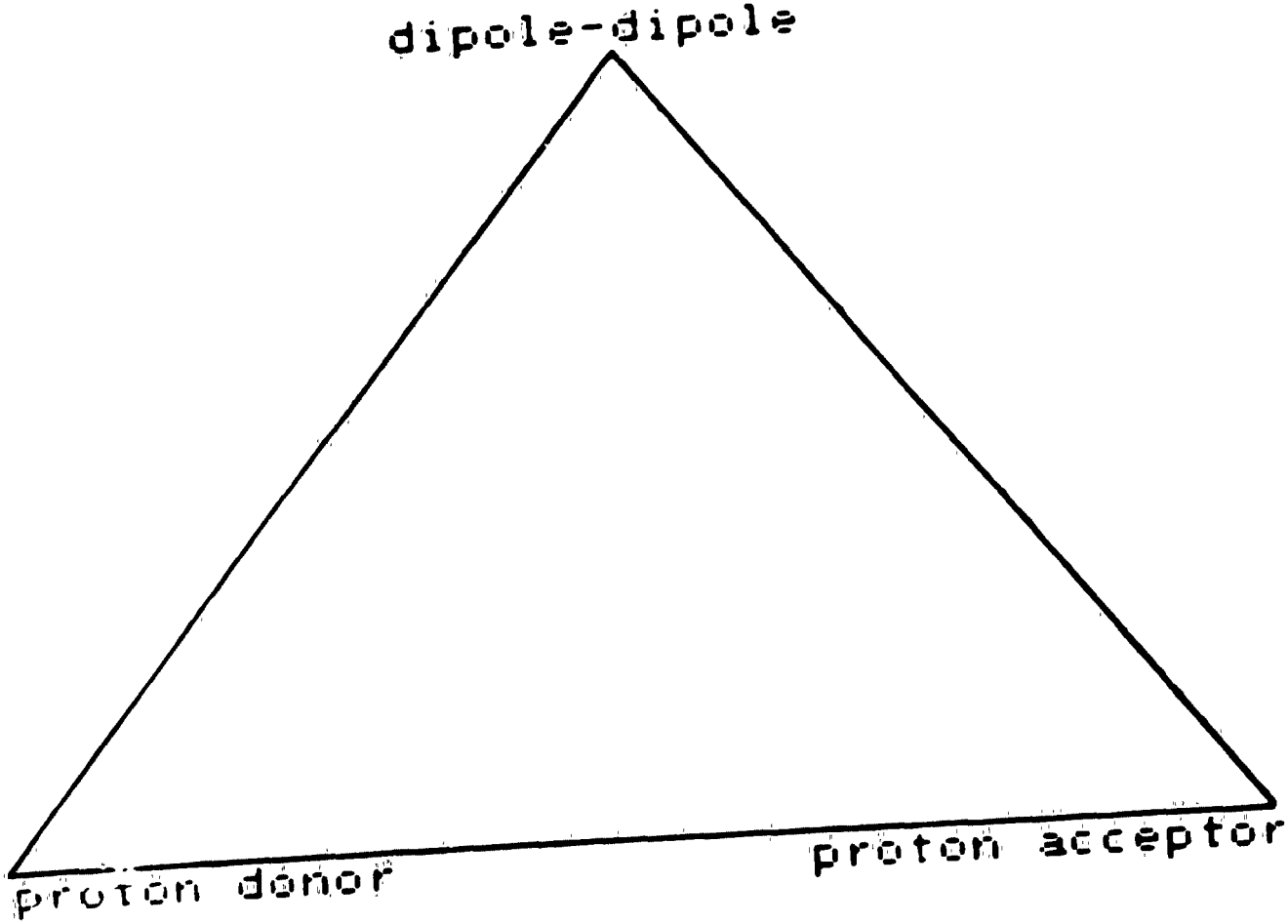

Figure 1. Interaction of solvents.

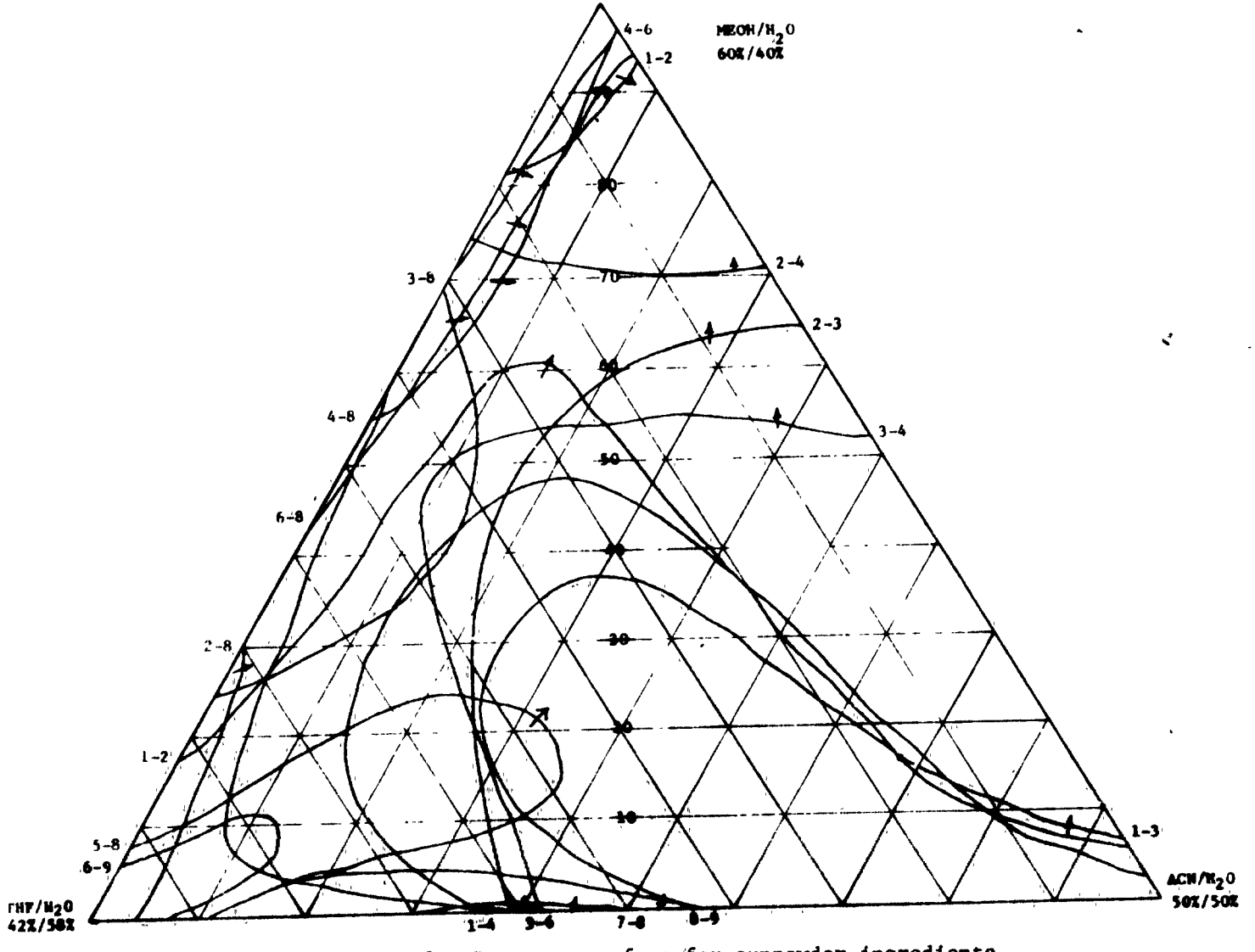

Figure 2. Response surface for gunpowder ingredients.

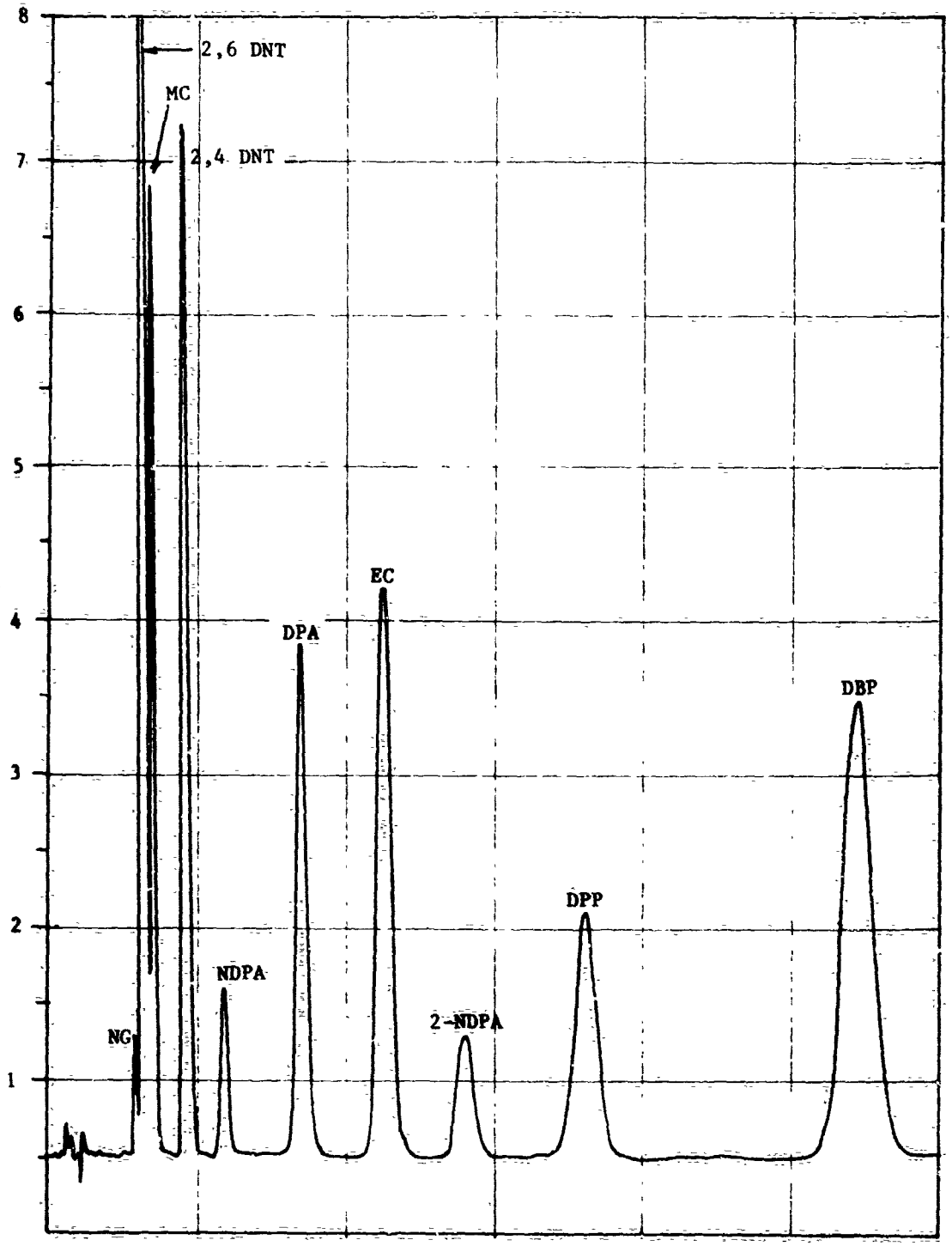

Figure 3. Chromatogram of mixture of ingredients.

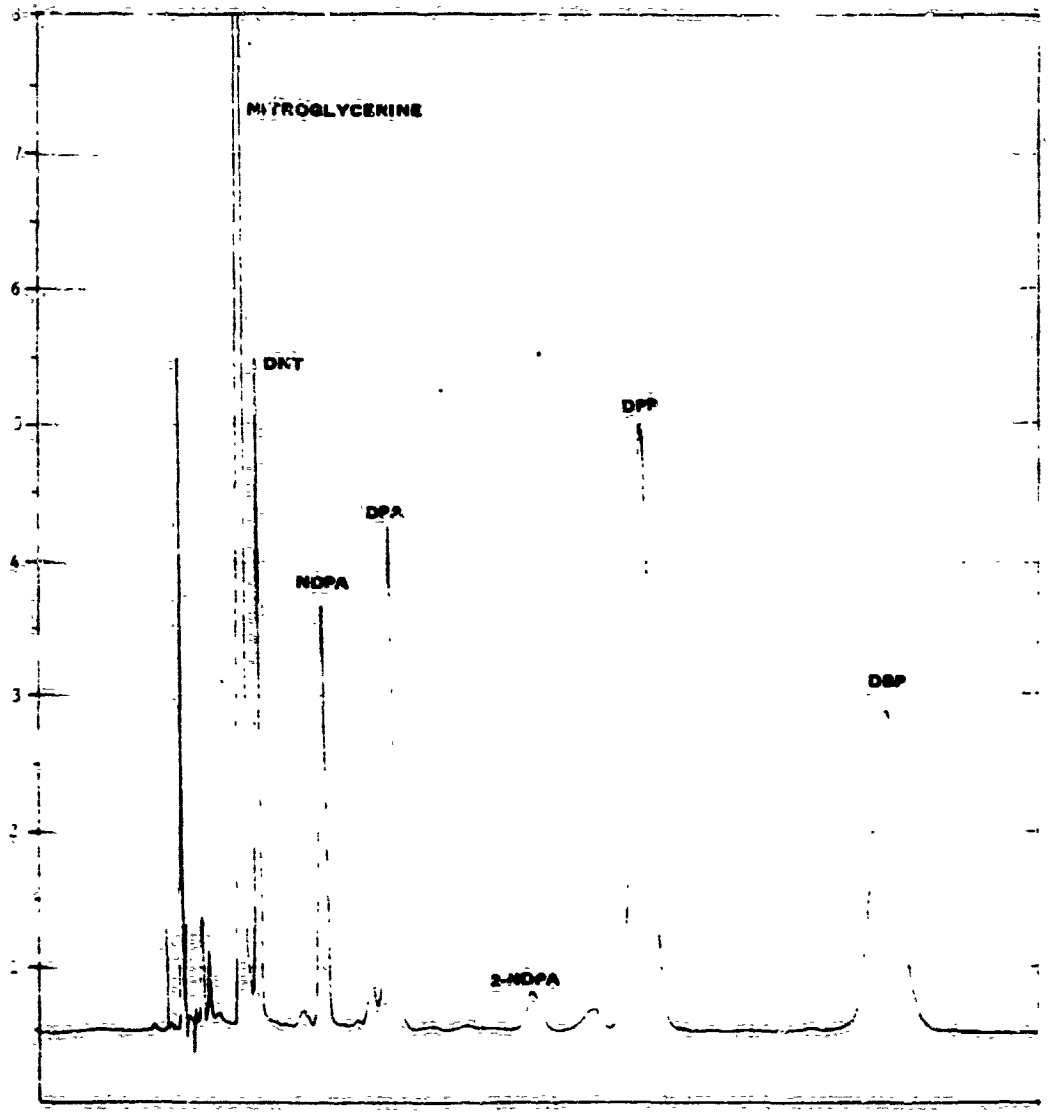

Figure 4. Gunpowder A analysis.

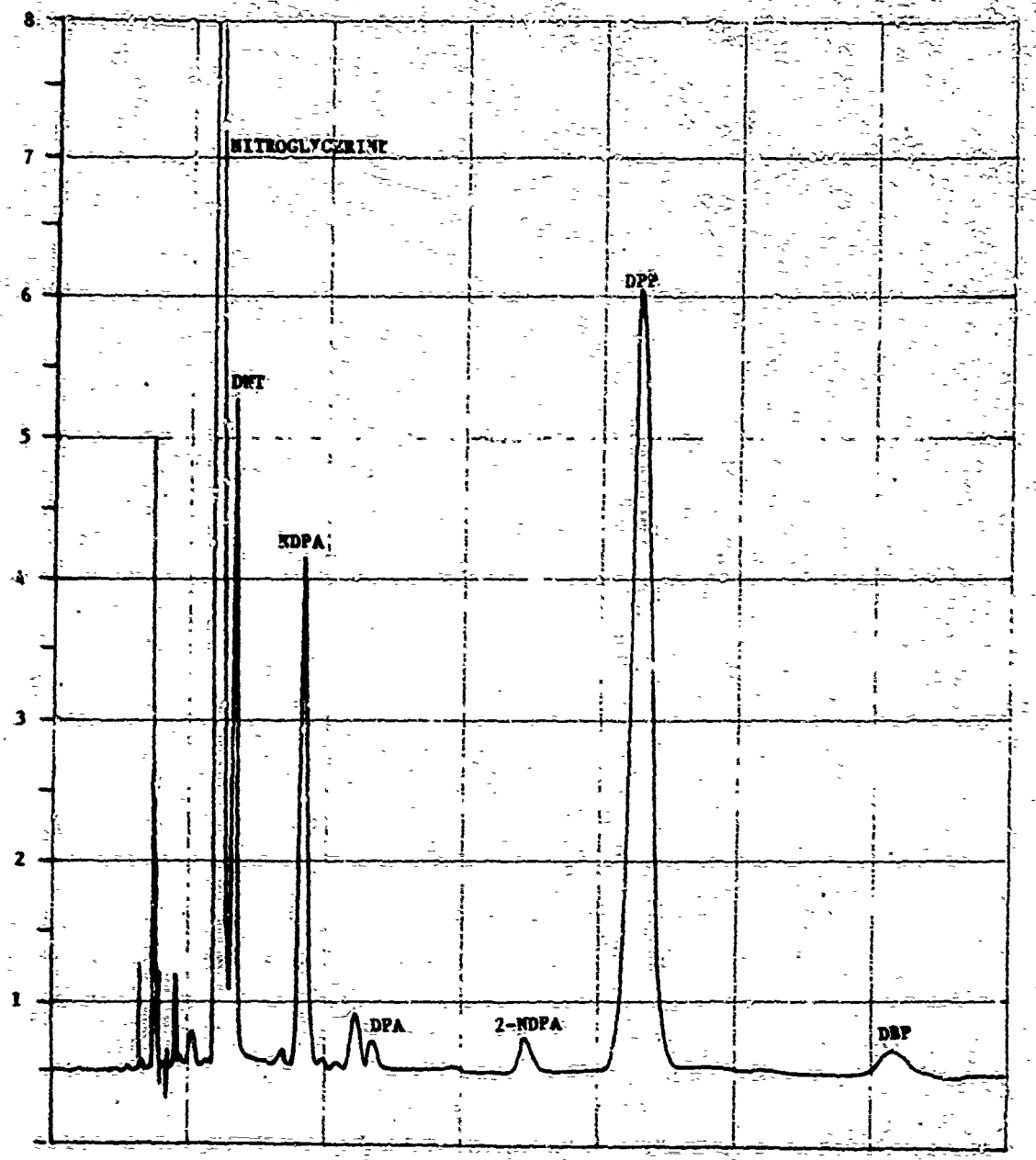

Figure 5. Gunpowder B analysis.

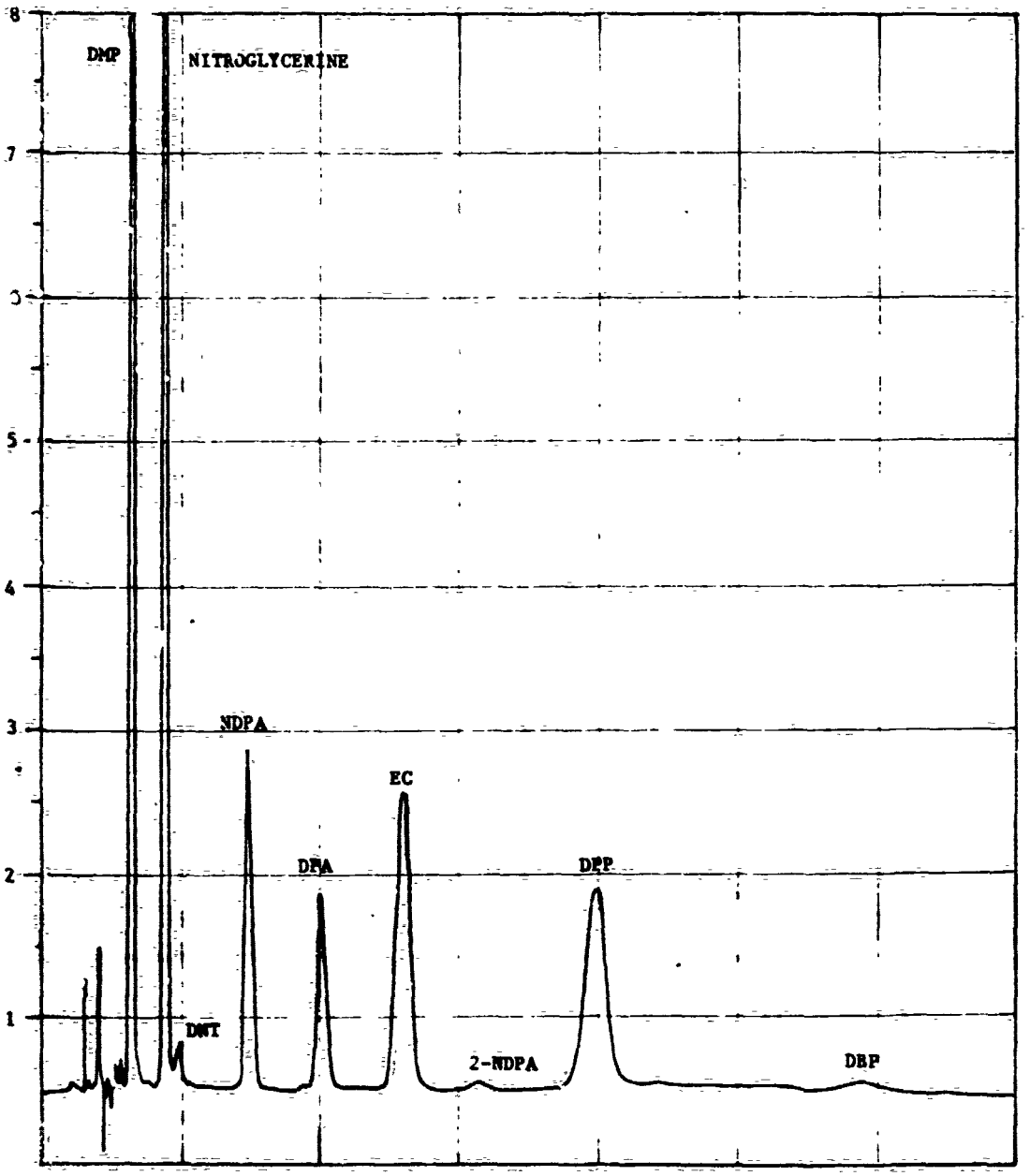

Figure 6. Gunpowder C analysis.

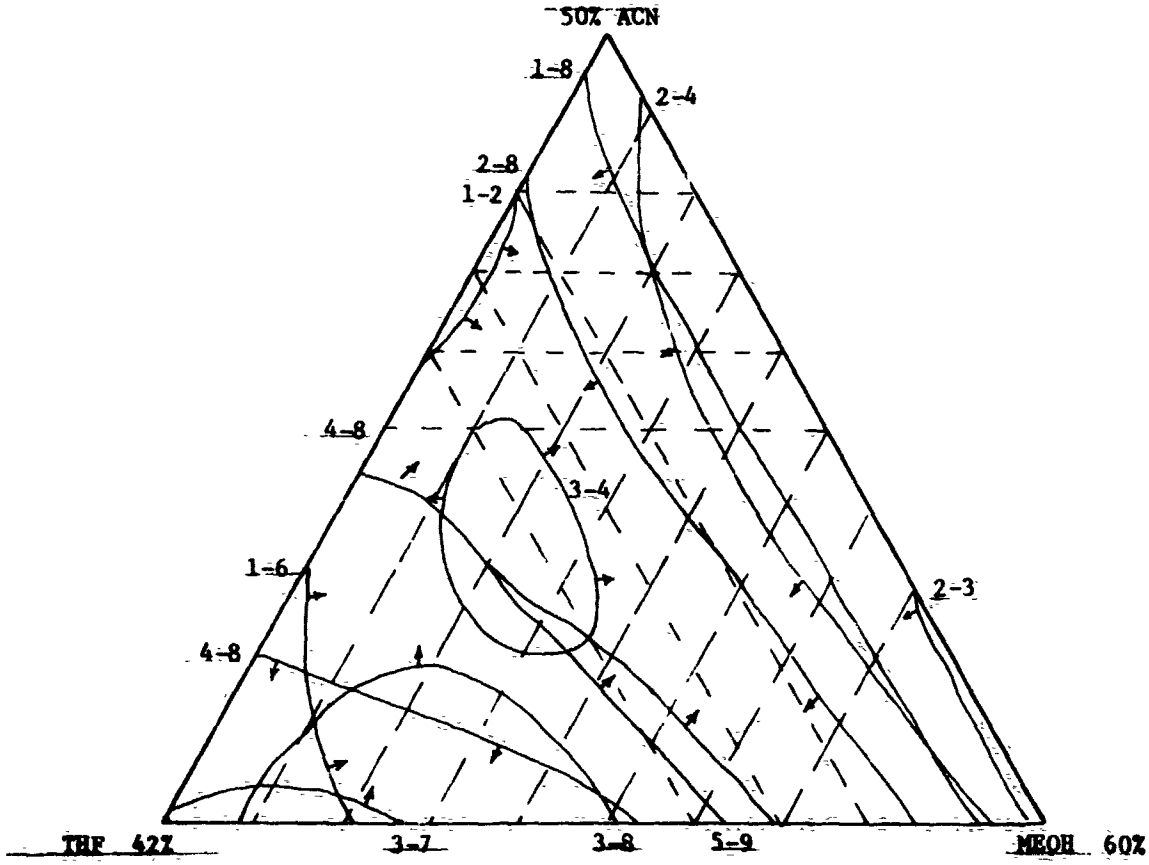

Figure 7. Response surface for propellant ingredients.

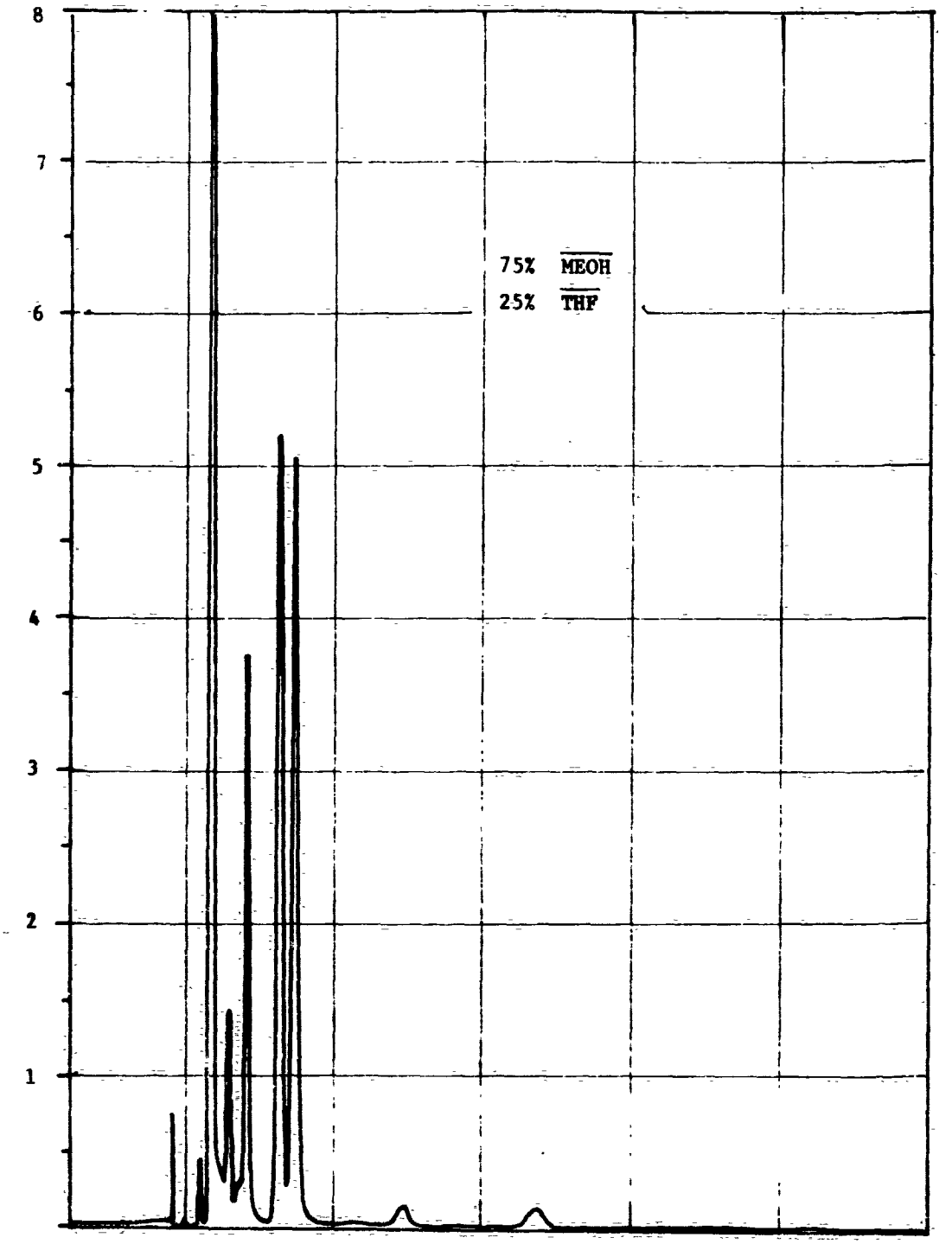

Figure 8. Chromatogram of propellant.

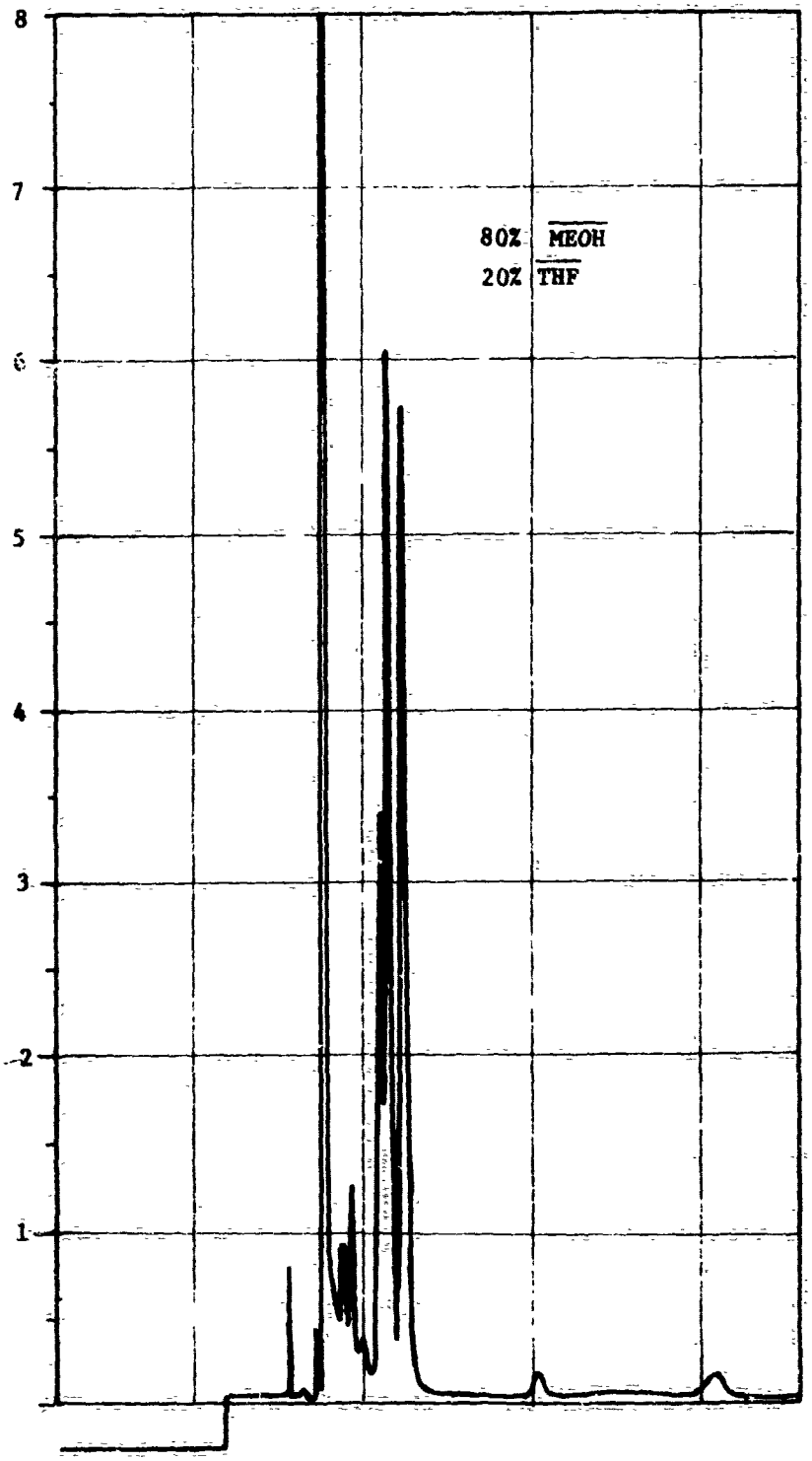

Figure 9. Chromatogram of propellant.

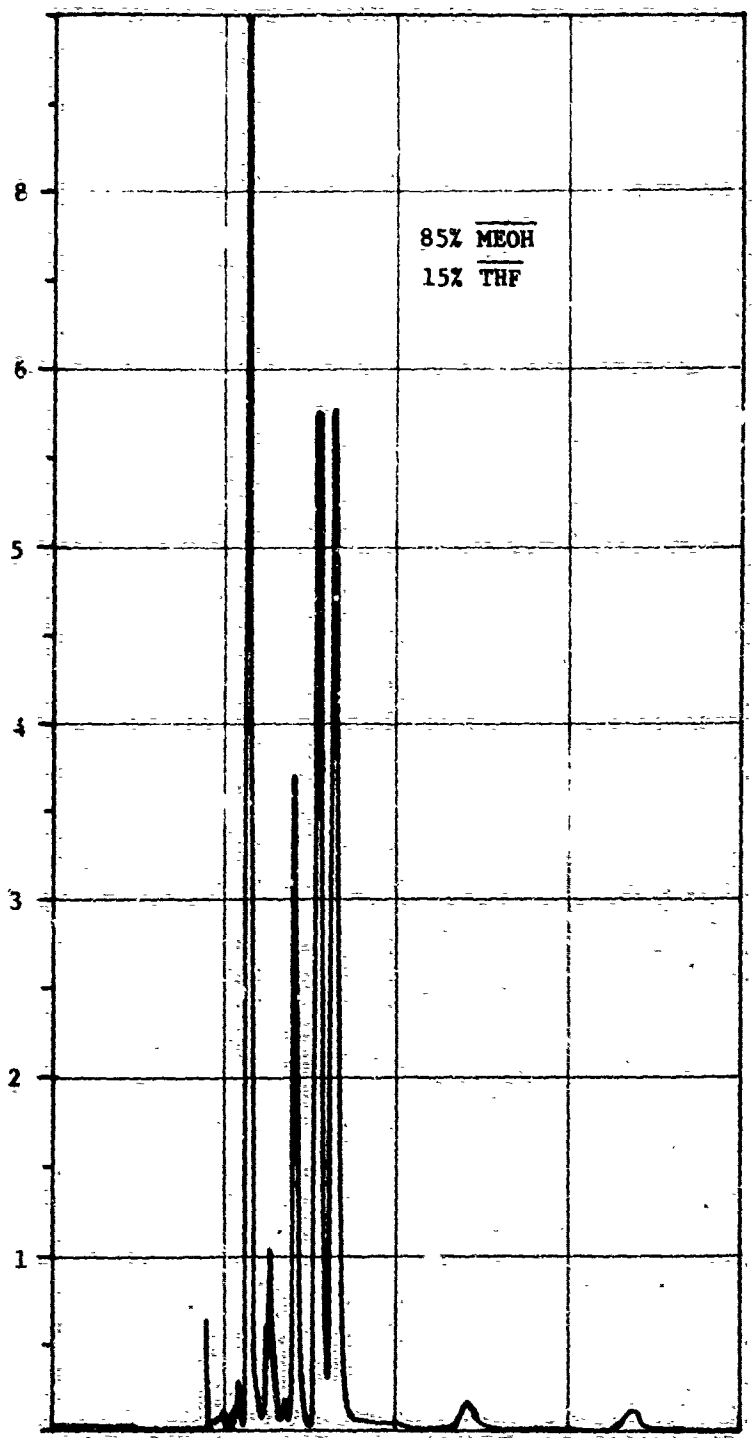

Figure 10. Chromatogram of propellant.

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